IEEE Nuclear Science Symposium Conference Record 91CH3100-5, vol. 1, pp. 143- 147, 1991.

X-RAY FLUORESCENCE MEASUREMENTS OF 412 INORGANIC COMPOUNDS*

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ABSTRACT

In a search for new, fast, inorganic scintillators, we have exposed 412 inorganic compounds to 0.5 ns pulses of 20 keV x-rays and measured their fluorescent emissions. Most of these compounds were dense (>4 g/cm^3), contained heavy cations such as Pb, Bi, Ba, or a rare-earth element, and anions such as O, F, Cl, Br, I, CO_3, SO_4, and PO_4. About half were undoped compounds and half contained 0.1% to 10% rare-earth dopants. Standard scintillators such as BaF_2 and BGO were included for reference. In this Conference Record we report total luminosities and fitted exponential decay times and percentages for 97 compounds having either a luminosity > 40% of BGO, or an initial photon intensity greater than BGO, or a component decay time <1 ns. Emission wavelengths are listed for 53 of these compounds. Significant fast emissions were seen from Y_2SiO_5(Ce), $CeCl_3$, $BaCl_2$, $PbSO_4$, and $LuPO_4$(Ce). Other less luminous compounds Yb_2O_3, CuI, CuBr, and $BiPO_4$(Gd) exhibited components with shorter decay times than ZnO(Ga) and BaF_2.

1 BACKGROUND

The background, motivation, and method for this search has been previously reported [1] (Figure 1). In summary, we seek scintillators able to detect energetic (> 400 keV) gamma rays with good full-energy detection efficiency, timing resolution, and light output. Our method exposes compounds in powdered form to pulses of synchrotron x-rays, permitting the testing of many more compounds than is possible using the traditional method of using gamma-ray sources and optical-quality crystals. The costly and time-consuming step of growing crystals can then be limited to the small fraction of compounds that appear promising in powdered form. Because this method is primarily sensitive to fluorescence from the surface of the powder, it can lead to "false positives," as some compounds absorb their own fluorescent emissions (radiation trapping) and therefore do not scintillate. Only after a crystal sample is shown to have fluorescent emissions whose intensity depends on the amount of ionizing energy deposited in the bulk of the crystal (e.g. a gamma-ray photopeak), is the compound established as a scintillator.

In previous work [1], we reported on the x-rayfluorescence properties of 85 compounds. In this work we expanded the number of samples to 412 and provide data on 97 of these compounds in this Conference Record.

2 METHOD

In this work, 412 compounds were exposed to 0.5 ns pulses of 20 keV monochromatic x-rays at the Brookhaven National Laboratory National Synchrotron Light Source (NSLS). Approximately 0.2 ml of each powder was placed in a cylindrical suprasil fused silica vial 5 mm in diameter with 0.37 mm wall thickness, and the tube was sealed with a plastic cap. A 20 keV x-ray beam is not significantly attenuated by the wall of the tube (attenuation length 1.2 mm). A Hamamatsu R2059 phototube with a fused silica window was used to detect any fluorescent emissions (Figure 1). Single photoelectrons were counted, and a pinhole was placed in front of the phototube for the more luminous compounds to reduce the detection rate to a small fraction of the 1.8 MHz x-ray excitation rate. The x-ray beam was aimed so that a portion missed the edge of the tube and was detected by a plastic scintillator and phototube detector which provided a trigger pulse [2] for the oscilloscope and the fluorescent decay time measurements described in the next section.

3 RESULTS

3.1 Fluorescent Luminosity

As described in reference [1], the total fluorescent luminosity was determined from the single photoelectron rate by correcting for counter deadtime, subtracting phototube background, adjusting for the synchrotron beam current, correcting for optical depth, and normalizing to a value of 1000 for BGO. Uncertainties in optical depth of the powdered samples prevented us from estimating the luminosity with an

* This work was supported in part by the U.S. Department of Energy under contracts DE-AC03-76SF00098 (LBL), DE-AC05-84OR214-00 (ORNL), and DE-AC02-76CH00016 (BNL); in part by Public Health Service Grants Nos. P01 25840 and R01 CA48002; in part by the Environmental Restoration and Waste Management Fellowship Program, and in part by The Whitaker Foundation.

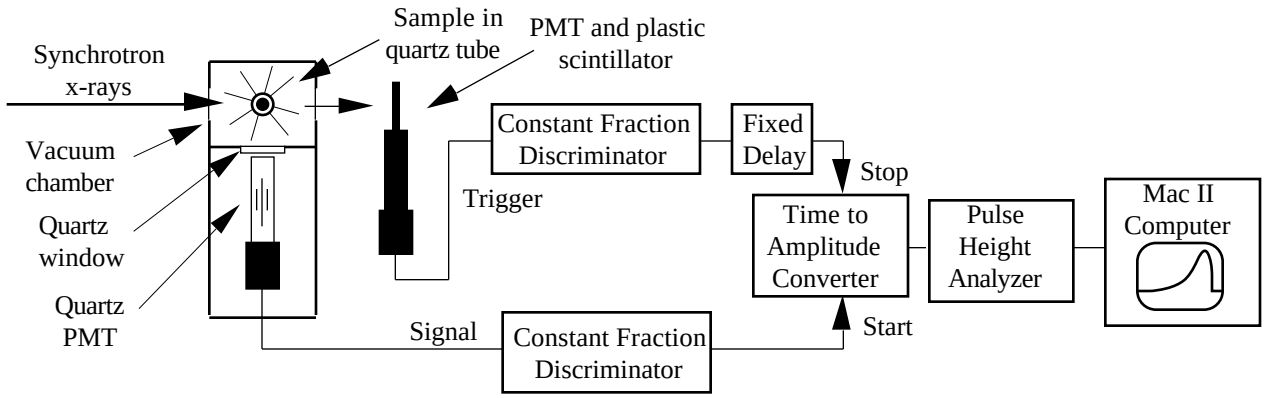

Figure 1. Experimental method for using synchrotron x-rays to measure the fluorescence decay timing spectrum of compounds in powdered form.

accuracy better than a factor of 3. We did not correct for the wavelength-dependent quantum efficiency of the phototube.

All purity data listed in Table 1 was provided by the manufacturers. In some cases the impurities could have significantly effected the observed emissions, either by acting as fluorescent emitters or by acting to quench the fluorescence.

3.2 Fluorescent Decay Times and Percentages

The fluorescence decay timing spectrum decay time was measured using the delayed coincidence method [3]. A single photoelectron pulse was used to start a time-to-amplitude converter (TAC). A delayed trigger pulse from the plastic scintillator stopped the TAC. A sum of up to three exponential decay components were fit to the time delay distribution as needed to achieve an acceptable chi squared (usually <1000 with 760 degrees of freedom). A constant background was added to make the total number of photons in the fit equal to the number measured. Since the beam repetition period was 560 ns, fitting to components with a decay time longer than 10 µs was usually not possible.

The best fit values for decay times and percentages were determined by varying all parameters to achieve a minimum χ^2 = K. Statistical uncertainties in the decay times and percentages were determined by finding the largest and smallest value of each parameter that gave a minimum χ^2 equal to K + 1 when that parameter was held fixed and all other parameters were varied. Typical uncertainties in fitted decay times ranged from 2% of the decay time for luminous components to over 20% of the decay time for weak components. Uncertainties in component intensities ranged from 0.1% to over 50%.

Table 1 lists luminosities, exponential decay times, and fractions for 97 compounds having either a luminosity > 40% of BGO, or an initial intensity >BGO, or a component decay time <1 ns. For doped compounds, percentages of dopants are by weight. For a list of all 150 compounds with an observed luminosity > 0.5% of BGO, their luminosity, fitted exponential decay times, percentages, and uncertainties, and a list of the remaining 260 compounds of lower luminosity, see reference [4].

3.3 Fluorescent Wavelengths

The emission spectra of 52 compounds were measured by placing a 0.125 meter Jarrell-Ash MonoSpec Monochromator between the sample and the quartz phototube (Table 2). The single photoelectron rate was recorded as the monochromator wavelength was scanned by computer control for two gratings, one blazed for 300 and the other blazed for 500 nm. The data were not corrected for the wavelength-dependent responses of either the monochromator or the phototube. For a more complete list of wavelengths, see reference [4].

4 Conclusions

Compounds with high atomic number and promising luminosity and speed include $PbSO_4$ (see reference [5]), $CeCl_3$ (brighter than BGO with 70% 23 ns), $BaCl_2$ (brighter than BGO with 36% 1.2 ns). Other less luminous compounds (such as CuI, Yb_2O_3, CuBr, and $BiPO_4$(Gd)) exhibited fluorescence components faster than ZnO(Ga) and BaF_2 (<0.5 ns). These compounds are interesting enough to prepare in single crystal form for further study.

5 Acknowledgements

We would like to thank Dr. Charles Bouldin of the National Institute of Standards and Technology for the use of beam line X23-A2 at the National Synchrotron Light Source. We thank Prof. Angela Stacey for assistance in locating uncommon inorganic chemicals and Angela Lai for handling and keeping track of all the chemicals used in this work. We thank Wilfred Lam for assistance in fitting the decay time curves. We thank R. Sparrow and D. Anderson for helpful discussions and R. Sparrow (Optovac, Inc.) and E. Flyckt (Philips, Holland) for providing chemicals.

IEEE Nuclear Science Symposium Conference Record 91CH3100-5, vol. 1, pp. 143- 147, 1991.

Table 1 Luminosity and fitted decay components of 97 compounds excited by 20 keV synchrotron x-radiation[a]

Formula	L[a]	Purity (%)	Color of powder	ρ (g/cc)	Exponential decay time (percentages)
Y_2SiO_5 + Ce[b,c]	17354		clear[d]	2.70	19.7 ns (32.4%), 50 ns (66.3%), >10 μs (1.3%)
$SnPO_3F$	8523		white	3.50	693 ns (14.6%), >10 μs (85.4%)
SrF_2[c]	4473		white	4.24	19.9 ns (2.0%), 408 ns (63.3%), >10 μs (34.8%)
Pilot U (plastic)	4056		white	1.03	1.5 ns (73.3%), 9.3 ns (11.0%), 97 ns (6.5%), >10 μs (9.2%)
$CeCl_3$[c]	3469	99.9	white	3.90	4.4 ns (6.6%), 23.2 ns (69.6%), 70 ns (7.5%), >10 μs (16.3%)
BaF_2[c]	3171		white	4.83	0.7 ns (5.3%), 66 ns (6.4%), 444 ns (82.9%), >10 μs (5.5%)
$BaCl_2$[c]	3002	tech	white	3.90	1.2 ns (36%), 3.5 ns (1.1%), 58 ns (24.5%), >10 μs (38.4%)
NE102 (plastic)	2555		white	1.03	1.9 ns (69%), 10.4 ns (11.9%), 108 ns (5.7%), >10 μs (13.4%)
$ScPO_4$ + 1% Tb_2O_3	2293	99.99	lt. yellow[d]	3.71	>10 μs (100%)
$ThCl_4$[c]	2142	99.9	lt. gray	4.59	24.5 ns (1.2%), 1.7 μs (76.6%), >10 μs (22.2%)
$SrCl_2$ + 0.3% Eu	2126	99.99	clear[d]	3.05	1.6 μs (100%)
$ScPO_4$ + 5% Eu_2O_3[c]	2080	99.99	clear[d]	3.71	2.7 ns (0.4%), 30 ns (2.3%), 380 ns (47%), >10 μs (50.3%)
SrI_2	1986	95	white	4.55	8.6 ns (1.5%), 53 ns (6.1%), 394 ns (92.5%)
$ScPO_4$ + 3% $^{161}Dy_2O_3$	1850	99.99	lt. pink[d]	3.71	5.9 ns (0.6%), >10 μs (99.4%)
SrO	1842	99.95	white	4.70	2.3 ns (0.9%), 80 ns (1.7%), >10 μs (97.4%)
$ScPO_4$ + 2% $^{163}Dy_2O_3$	1761	99.99	clear[d]	3.71	3.7ns (0.4%), 30.8 ns (2.4%), 339 ns (43.6%), >10 μs (53.7%)
ZrO_2	1482	99+	white	5.60	1.01 μs (33.2%), >10 μs (66.8%)
$CaWO_4$	1325		white	6.06	20 ns (0.4%), 3.6 μs (99.6%)
YPO_4 + 1% Tb[c]	1209	99.99	clear[d]	4.31	>10 μs (100%)
YPO_4 + 0.6% $^{161}Dy_2O_3$[c]	1122	99.99	lt. yellow[d]	4.31	>10 μs (100%)
$CaMoO_4$	1036	99.9+	white	4.40	8.4 μs (100%)
$ScPO_4$ + 2% Yb_2O_3[c]	1024	99.99	clear[d]	3.71	21.1 ns (81.9%), 122 ns (2.3%), >10 μs (15.1%)
$Bi_4Ge_3O_{12}$ (BGO)[c]	1000	99.99995	white	7.13	4.6 ns (1.1%), 45.5 ns (7.8%), 277 ns (90.3%)
$ScPO_4$ + 10% Sm_2O_3[c]	993	99.99	lt. yellow[d]	3.71	6.3 ns (2.0%), 36.3 ns (8.7%), >10 μs (89.3%)
YPO_4 + 9% $^{163}Dy_2O_3$	983	99.99	lt. yellow[d]	4.31	>10 μs (100%)
YPO_4 + 1% Dy_2O_3[c]	963	99.99	lt. yellow[d]	4.31	>10 μs (100%)
YPO_4 + 0.7% $^{162}Dy_2O_3$	884	99.99	lt. yellow[d]	4.31	>10 μs (100%)
$ZnWO_4$	869	98	white	7.87	936 ns (11.3%), >10 μs (88.7%)
$SrCl_2$ + 0.2% Lu[c]	862	99.99	clear[d]	3.05	9.2 ns (0.3%), 485 ns (87.7%), >10 μs (12.1%)
$LuPO_4$ + 0.6% $^{161}DyO_3$	820	99.99	clear[d]	6.53	13.6 ns (0.2%), >10 μs (99.8%)
YPO_4 + 2% Tm_2O_3	815	99.99	lt. yellow[d]	4.31	>10 μs (100%)
$ScPO_4$ + 3% Sm_2O_3	797	99.99	clear[d]	3.71	1.2 ns (0.2%), 26.2 ns (5.8%), 75 ns (14.4%), >10 μs (79.5%)
$SrCl_2$ + 0.2% Co	777	99.99	clear[d]	3.05	3.7 ns (0.2%), 496 ns (94.0%), >10 μs (5.8%)
$ScPO_4$ + 2% Nd_2O_3[c]	724	99.99	clear[d]	3.71	3.4 ns (0.3%), 62 ns (11.2%), 404 ns (35.2%), >10 μs (53.3%)
$LuPO_4$ + 1% Ce[c]	591	99.99	clear[d]	6.53	5.1 ns (4.0%), 23 ns (87.6%), >10 μs (8.4%)
$ScPO_4$ + 2% $^{145}Nd_2O_3$[c]	578	99.99	clear[d]	3.71	3.8 ns (0.4%), 48.7 ns (10.0%), 152 ns (36.3%), >10 μs (53.4%)
$MgWO_4$	571	99	white	5.66	>2 μs (100%)
$LuPO_4$ + 0.6% $^{163}Dy_2O_3$	547	99.99	clear[d]	6.53	>8 μs (100%)
$LuCl_3$[c]	541	99.9	white	3.98	4.5 ns (2.1%), 28.5 ns (3.9%), 254 ns (25.2%), >10 μs (68.7%)
$PbSO_4$[c]	517	99.999	white	6.20	7.4 ns (7.2%), 33.7 ns (21.3%), 168 ns (45.9%), >10 μs (25.7%)
$TlPF_6$[c]	496		white	4.63	<0.6 ns (0.4%), 26 ns (2.7%), 399 ns (74.4%), >10 μs (22.5%)
$LuPO_4$ + 0.25% Dy_2O_3	474	99.99	clear[d]	6.53	>10 μs (100%)
CeF_3	436		white	6.16	4.2 ns (31.8%), 23.6 ns (55.8%), >10 μs (12.4%)
$ScPO_4$ + 0.7% VO_2[c]	431	99.99	clear[d]	3.71	3.6 ns (0.9%), 26.2 ns (5.0%), 103 ns (61.3%), >10 μs (32.9%)
$ScPO_4$ + 2% NiO[c]	413	99.99	clear[d]	3.71	5.9 ns (1.6%), 36.8 ns (7.1%), 98 ns (59.4%), >10 μs (31.9%)
$La_2(ZrO_3)_3$	404		white	5.00	2.6 ns (0.4%), 94 ns (3.3%), >10 μs (96.3%)
ZnO + Ga[c]	396		lt. gray	5.61	0.6 ns (97.5%), >10 μs (2.5%)
YPO_4 + 5% Nd_2O_3[c]	364	99.99	lt. yellow[d]	4.31	10.3 ns (15.4%), 27 ns (30.3%), 142 ns (24.9%), >10 μs (29.4%)

[a] Luminosity in the 160- 580 nm wavelength range, corrected for optical depth, and normalized to BGO = 1000.
[b] P47 phosphor
[c] Wavelengths given in Table 2
[d] small crystals

Table 1 (continued) Luminosity and fitted decay components of 97 compounds excited by 20 keV synchrotron x-radiation[a]

Formula	L[a]	Purity (%)	Color of powder	ρ (g/cc)	Exponential decay times (percentages)
Y_2O_3[c]	354		white	5.00	4.9 ns (6.8%), 22.5 ns (26.9%), 62 ns (62.8%), >10 μs (3.6%)
LuF_3[c]	346		white	8.32	0.8 ns (1.0%), 43.9 ns (2.3%), >10 μs (96.7%)
YF_3[c]	285	99.9	white	4.01	0.7 ns (1.5%), 21.3 ns (1.4%), 576 ns (16.9%), >10 μs (80.2%)
ZnO[c]	253		clear[d]	5.61	0.5 ns (0.7%), 1.48 μs (57.6%), >10 μs (41.7%)
Ga_2O_3	251	99.999	white	5.88	1.6 ns (8.0%), 14.5 ns (14.5%), 112 ns (22.0%), >10 μs (55.6%)
$RbCaF_3$[c]	226	99.99	clear[d]	3.43	1.3 ns (21.8%), 2.8 ns (31.3%), 13.1 ns (1.6%), >10 μs (45.4%)
$Tb_2(SO_4)_3$	203	99.9	white	5.00	<0.5 ns (0.2%), >10 μs (99.8%)
TbF_3[c]	200	99.9	white	7.23	0.7 ns (0.3%), >10 μs (99.7%)
YPO_4 + 2% Pr_2O_3[c]	191	99.99	lt. yellow[d]	4.31	8.7 ns (24.3%), 20.5 ns (14.9%), >10 μs (60.8%)
$BaHPO_4$[c]	183		white	4.10	0.6 ns (5.7%), 75 ns (11.6%), 545 ns (82.7%)
$AgBF_4$[c]	160	99	off-white	4.37	<0.5 ns (1.4%), 126 ns (6.3%), >10 μs (92.3%)
$SnSO_4$	152	95+	white	4.28	<0.5 ns (0.6%), 25.4 ns (0.9%), >10 μs (98.5%)
ZnO[c]	133	99.9+	white	5.67	<0.5 ns (4.8%), 8 ns (1.8%), 224 ns (14.6%), >10 μs (78.8%)
CdI_2[c]	106	99	white	5.67	1.4 ns (45.6%), 6 ns (29.9%), 45.2 ns (14.2%), >10 μs (10.3%)
Eu_2O_3	99		pale rose	7.42	<0.5 ns (0.3%), >10 μs (99.7%)
Rb_2CO_3[c]	96	99.9	white	3.40	<0.5 ns (2.2%), >10 μs (97.8%)
CuI[c]	73	98	off-white	5.62	<0.5 ns (88%), >10 μs (12%)
$ZrSiO_4$	72	98	white	4.56	<0.6 ns (0.7%), 14.6 ns (2.7%), 140 ns (23.5%), >10 μs (73.1%)
EuF_3[c]	72	99+	white	6.50	0.6 ns (0.7%), >10 μs (99.3%)
$2(PbCO_3)_2 \cdot Pb(OH)_2$[c]	64	99.999	white	6.14	4.3 ns (24.5%), 16.9 ns (37.1%), 73 ns (29.6%), >10 μs (8.7%)
$CdBr_2$	63	99	white	5.19	<0.5 ns (6.8%), 5.7 ns (11.8%), 24 ns (12.9%), >10 μs (68.5%
$PbWO_4$[c]	60	99.998	white	8.23	1.6 ns (27%), 9.3 ns (35.9%), 37 ns (22.8%), >10 μs (14.3%)
$RbMgF_3$[c]	55	99.99	clear[d]	4.30	<0.5 ns (1.2%), 13.1 ns (0.4%), >10 μs (98.3%)
$AgNO_2$	48	99	white	4.45	<1 ns (1.0%), >10 μs (97.7%)
TaF_5	40	99	off-white	4.74	<0.8 ns (1.2%), 19.3 ns (3.7%), 134 ns (13%), >10 μs (82.1%)
$PbCO_3$[c]	38	99.999	white	6.60	2.9 ns (30.9%), 15.1 ns (37.3%), 80 ns (17.9%), >10 μs (13.9%)
$ZnMoO_4$	35	98+	off-white	5.63	0.9 ns (0.7%), >10 μs (99.3%)
$CdCl_2$	34	99+	white	4.00	<0.5 ns (3.3%), 21.3 ns (22.3%), 1.09 μs (74.5%)
PbF_2 + 0.5% Tb[c]	31		white	8.24	<0.5 ns (1.1%), >10 μs (98.9%)
$PbCl_2$[c]	31		white	5.90	2.0 ns (35%), 19.5 ns (19.4%), >10 μs (45.6%)
$BaBr_2$[c]	30		white	4.78	<0.5 ns (2.9%), 28.5 ns (0.8%), >10 μs (96.3%)
Lu_2O_3	28	99.9	white	9.42	0.8 ns (9.5%), 25.2 ns (4.8%), 302 ns (30.7%), >10 μs (55%
$3(Zn(OH)_2) \cdot 2(ZnCO_3)$	24		white	4.40	0.6 ns (3.7%), >10 μs (96.3%)
$SrZrO_3$	24	95	white	5.46	0.8 ns (0.7%), 17.6 ns (6.9%), 183 ns (20.5%), >10 μs (71.9%)
ZnF_2[c]	24		white	4.95	0.9 ns (7.2%), 10.3 ns (10.6%), 86 ns (15.3%), >10 μs (66.9%)
PbF_2 + 2% TbF_3	20		white	8.24	0.5 ns (1.6%), >10 μs (98.4%)
$Dy_2(SO_4)_3$	19	99.9	white	3.75	<0.5 ns (2.0%), 473 ns (23%), >10 μs (75%)
$Eu_2(WO_4)_3$[c]	15		white	7.37	<0.5 ns (0.9%), 77 ns (3.3%), >10 μs (95.8%)
$Tb_2(CO_3)_3 \cdot X(H_2O)$[c]	14	99.9	white	6[e]	<0.6 ns (1.6%), 42.2 ns (1.0%), >10 μs (97.4%)
Yb_2O_3[c]	13	99.9	white	9.17	<0.5 ns (72.7%), >10 μs (27.3%)
YPO_4	13	99.9	white	4.30	<0.6 ns (2.4%), 282 ns (7.4%), >10 μs (90.2%)
$La_2(WO_4)_3$	13		lt. green	6.63	<0.5 ns (1.3%), 279 ns (72.1%), >10 μs (26.6%)
TlCl	12	99.999	white	7.00	0.6 ns (25.9%), 3.4 ns (37.7%), 11.2 ns (18.2%), >10 μs (18.2%)
CuBr	11	99	off-white	5.00	<0.6 ns (43.4%), 6.4 ns (18.7%), >10 μs (37.9%)
ErF_3[c]	8	99.99	lt. pink	7.81	0.6 ns (4.3%), 42 ns (17.3%), >10 μs (78.4%)
$PbBr_2$	6	99.999	white	6.66	0.9 ns (13.6%), 8.3 ns (20.2%), 69 ns (23.2%), >10 μs (43%)
$Pb_2P_2O_7$[c]	4	99.99	clear[d]	5.80	1.1 ns (17.4%), 7 ns (20.1%), 75 ns (23.5%), >10 μs (39.0%)
$BiPO_4$ + 1% Gd[c]	2		white	6.32	<0.6 ns (21.4%), 3.3 ns (15.6%), 20.4 ns (8.5%), >10 μs (54.5%)

[a] Luminosity in the 160- 580 nm wavelength range, corrected for optical depth, and normalized to BGO = 1000.
[c] Wavelengths given in Table 2
[d] small crystals
[e] Density unavailable- value shown was assigned solely to permit calculation of luminosity

IEEE Nuclear Science Symposium Conference Record 91CH3100-5, vol. 1, pp. 143- 147, 1991.

Table 2 Fluorescence Emission Wavelengths[a]

Formula	Wavelengths (nm)	Formula	Wavelengths (nm)
$AgBF_4$	240, 330	$ScPO_4$ + 2% $^{145}Nd_2O_3$	220, 290, 320, 360, 390, 480, 540
$BaBr_2$	400	$ScPO_4$ + 2% Nd_2O_3	220, 290, 320, 360, 390, 480, 540
$BaCl_2$	300	$ScPO_4$ + 2% NiO	220, 310, 380, 480
BaF_2	220, 310	$ScPO_4$ + 2% Yb_2O_3	280, 370
$BaHPO_4$	320	$ScPO_4$ + 5% Eu_2O_3	360, 620
$Bi_4Ge_3O_{12}$ (BGO)	480	$ScPO_4$ + 10% Sm_2O_3	210, 310, 570
$BiPO_4$ + 1% Gd	310	$SrCl_2$ + 0.2% Lu	370
CdI_2	500	SrF_2	300
$CeCl_3$	360	SrI_2	510
CuI	430	$Tb_2(CO_3)_3 \cdot X(H_2O)$	490, 550
ErF_3	320, 380	TbF_3	490, 550
$Eu_2(WO_4)_3$	620	$ThCl_4$	340
EuF_3	590	$TlPF_6$	210, 320, 370
$LuCl_3$	380, 580	Y_2O_3	340
LuF_3	270, 310	Y_2SiO_5 + Ce	440
$LuPO_4$ + 1% Ce	340, 360	Yb_2O_3	350
$2(PbCO_3)_2 \cdot Pb(OH)_2$	470	YF_3	310, 380, 410, 480, 570
$Pb_2P_2O_7$	310	YPO_4 + 0.6% $^{161}Dy_2O_3$	360, 480, 580
$PbCl_2$	500	YPO_4 + 1% Dy_2O_3	240, 480, 580
$PbCO_3$	480	YPO_4 + 1% Tb	380, 410, 490, 550, 590
PbF_2 + 0.5% Tb	360, 380, 490, 540	YPO_4 + 2% Pr_2O_3	240, 270, 470, 490, 600
$PbSO_4$	350	YPO_4 + 5% Nd_2O_3	190, 240, 280
$PbWO_4$	460	ZnF_2	340
Rb_2CO_3	360	ZnO + Ga	390
$RbCaF_3$	240, 280	ZnO	390, 510
$RbMgF_3$	290		
$ScPO_4$ + 0.7% VO_2	210, 480		

[a] Wavelength data near 430 nm contaminated by a light leak

This work was supported in part by the Director, Office of Energy Research, Office of Health and Environmental Research, Medical Applications and Biophysical Research Division of the U.S. Department of Energy under contract No. DE-AC03-76SF000098, and in part by Public Health Service Grant Nos. P01 HL25840 and R01 CA48002 awarded by the National Heart Lung and Blood and National Cancer Institutes, Department of Health and Human Services. Research carried out at the National Synchrotron Light Source, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Division of Materials Sciences and Division of Chemical Sciences under contract No. DE-AC02-76CH00016. Research at Oak Ridge National Laboratory sponsored by the Division of Materials Sciences, U. S. Department of Energy under contract No. DE-AC05-84-OR214-00 with Martin Marietta Energy Systems, Inc.

One of us (T. DeVol) performed this research under appointment to the Environmental Resoration and Waste Management Fellowship Program, which is administered by Oak Ridge Associated Universities for the U.S. Department of Energy, and one of us (W.W. Moses) was supported in this research by the Whitaker Foundation.

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